



# MULTI-ELEMENT CONTENT CHARACTERIZATION OF COLD PRESS EDIBLE OILS PRODUCED FROM TWELVE SUNFLOWER VARIETIES

Ivan Donev<sup>1</sup>, Biljana Balabanova<sup>1</sup>, Sasa Mitrev<sup>1</sup>

<sup>1</sup>Faculty of Agriculture, Goce Delčev University, , Krste Misirkov No. 10-A, 2000 Stip, Republic of Macedonia

## INTRODUCTION

The content of the following isotopes of the 36 elements (Li<sup>7</sup>, Be<sup>9</sup>, B<sup>11</sup>, Na<sup>23</sup>, Mg<sup>24</sup>, Al<sup>27</sup>, P<sup>31</sup>, Ca<sup>39</sup>, Ti<sup>48</sup>, V<sup>51</sup>, Cr<sup>53</sup>, Mn<sup>55</sup>, Fe<sup>56</sup>, Co<sup>59</sup>, Ni<sup>60</sup>, Cu<sup>63</sup>, Zn<sup>64</sup>, Ga<sup>71</sup>, Ge<sup>74</sup>, As<sup>75</sup>, Se<sup>77</sup>, Rb<sup>85</sup>, Sr<sup>88</sup>, Mo<sup>95</sup>, Pd<sup>106</sup>, Ag<sup>107</sup>, Cd<sup>111</sup>, In<sup>115</sup>, Sn<sup>120</sup>, Sb<sup>121</sup>, Cs<sup>133</sup>, Ba<sup>137</sup>, Tl<sup>205</sup>, Pb<sup>206/207/208</sup> and Bi<sup>209</sup>) in EDIBLE OILS produced from TWELVE SUNFLOWER VARIETIES from Republic of Macedonia were determined.

## QA/QC

### Standard addition method:

10 ppb; R (88.1-112%)  
50 ppb; R (85.1-102%)  
150 ppb; R (92-105%)  
1 ppm; R (75.1-114%)

### Certified reference material

CRM-TMSO (metals in soybean oil)  
(High-purity standards, Charleston, CS, USA)  
For: Ag, Ca, Cu, Fe, Mg, Ni, P, Pb, Zn

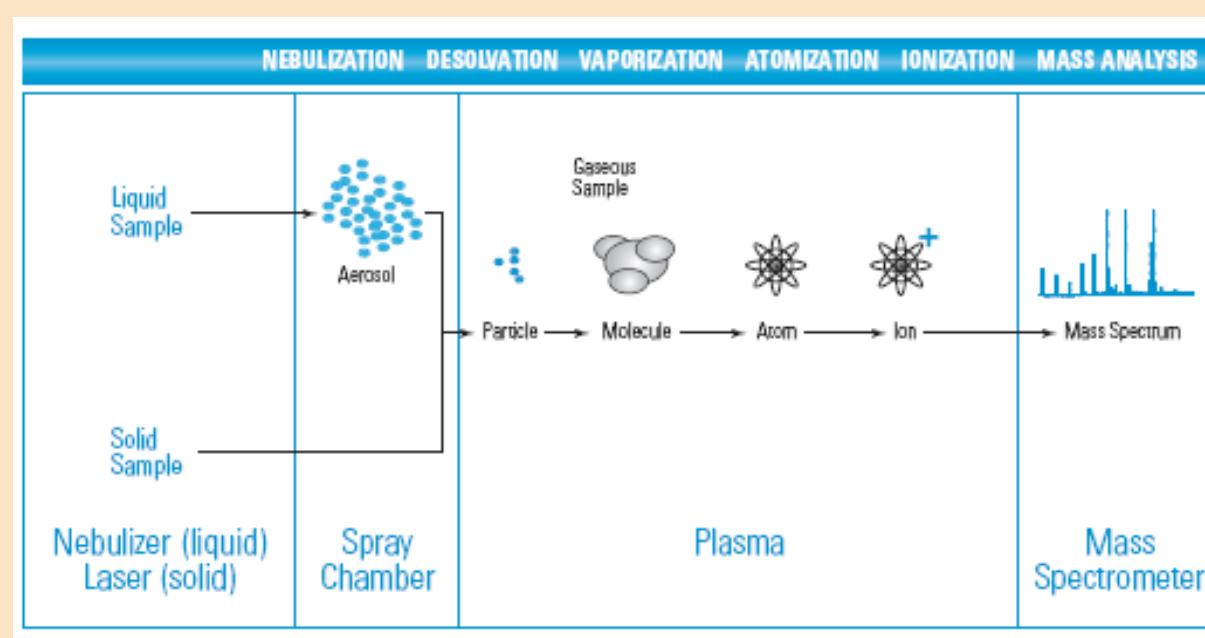
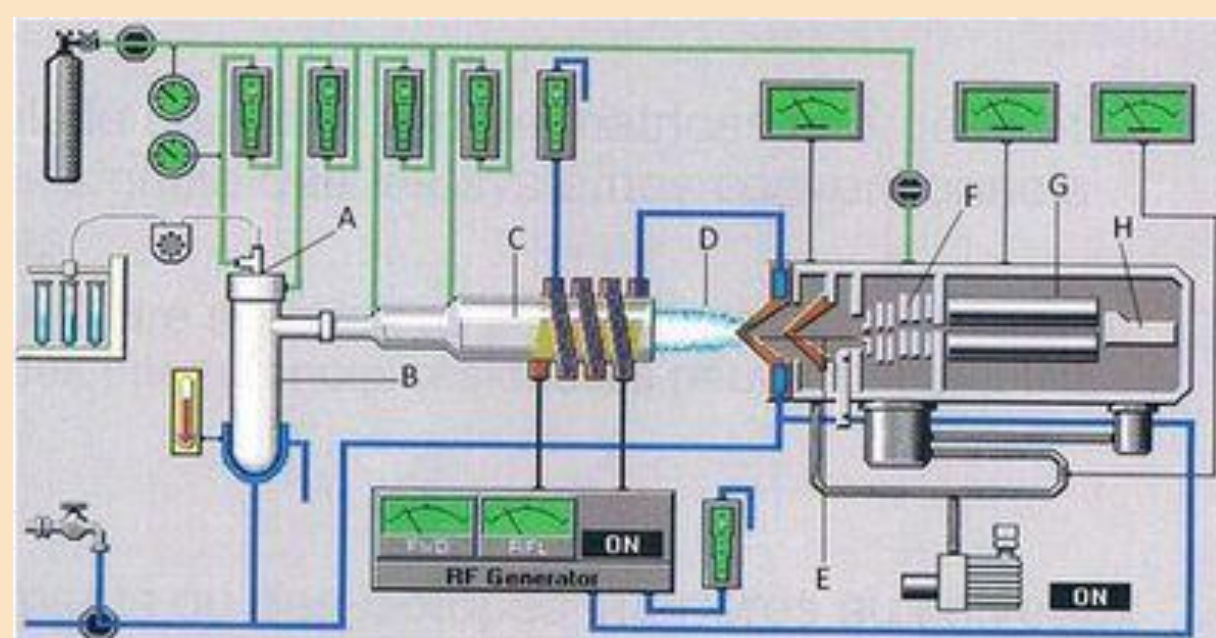
**Recoveries ranges in 87.5-109%**

**t- test (p=0.005)**  
**no-significant differences were found**

## ANALYTICAL METHODS



Inductively-coupled plasma-mass spectrometry (ICP-MS) has been used for determination of the elements content, after microwave digestion, employing nitric acid and hydrogen peroxide in this step. The method has been validated using both an oil reference material and recovery experiments over different oil samples, obtaining satisfactory results in both cases. Interday repeatability lower than 10% was observed for all of the analyzed elements in the analyzed oil samples.

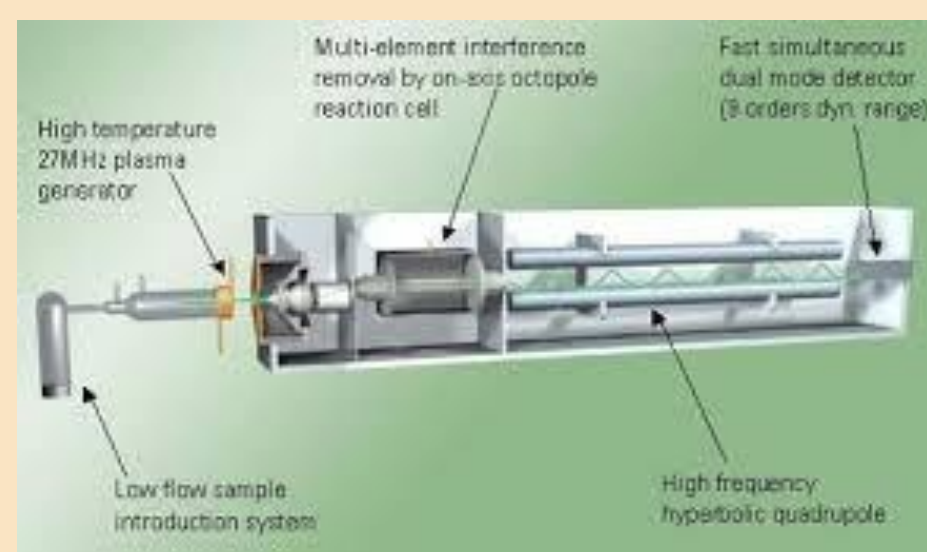


INSTRUMENT	(ICP introduction system)
Sampler	Cu (standard)
Skimmer	Ni (standard)
Nebulizer	MicroMist (standard)
Plasma torch	Quartz, 2.5 mm (standard)
<b>Integration Time</b> (for all analyzed elements)	0.3 sec x 1 point
Replication	3
<b>Tune parameters</b>	
RF power	1500 W
Sample depth	8.5 mm
Carrier gas	0.80 L/min
Makeup gas	0.23 L/min
Extract lens 1	-3 V
Extract lens 2	-150 V
Energy discriminator	2 V
Reaction gas	He 5.0 mL/min
CeO/Ce	0.58% (ref. value <0.65%)
Ce <sup>++</sup> /Ce	2.05% ((ref. value <3%)

**INTERFERENCES** in ICP-MS - MOLECULAR (POLYATOMIC) ion at the same nominal mass as the isotope of interest

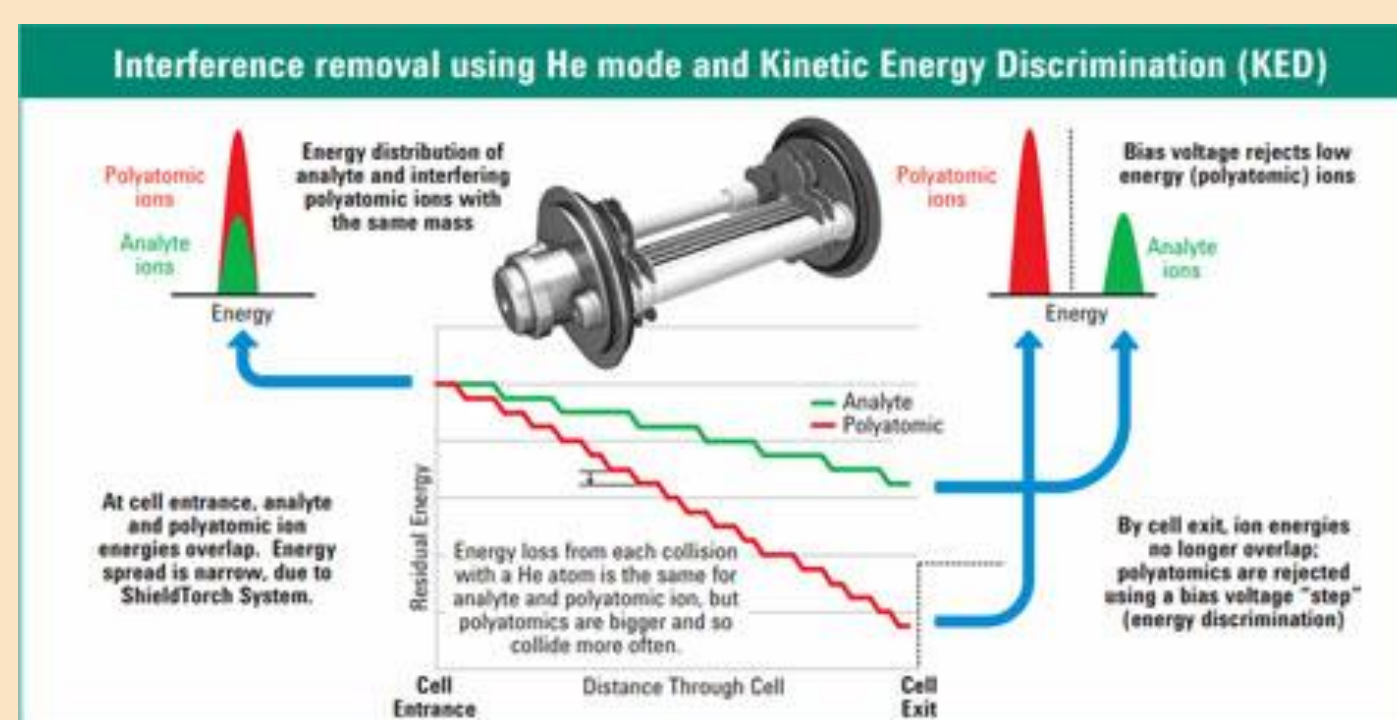
(plasma-based), such as <sup>40</sup>Ar, <sup>40</sup>Ar<sup>16</sup>O, and <sup>40</sup>Ar<sup>38</sup>Ar

(matrix-based), such as <sup>35</sup>Cl<sup>16</sup>O, and <sup>32</sup>S<sup>32</sup>S



## COLLISION / REACTION CELL (CRS) with optional He gas

<sup>51</sup>V, <sup>53</sup>Cr, <sup>57</sup>Fe, <sup>60</sup>Ni, <sup>63</sup>Cu, <sup>66</sup>Zn, <sup>69</sup>Ga, <sup>72</sup>Ge, <sup>75</sup>As, <sup>77</sup>Se



## CONDUCTED INVESTIGATIONS/PERSPECTIVES

Table 1. Basic statistics for elements contents in edible oils

Element	B	Na	Mg	Si	Al	P	K	Ca	Fe	Cu	Zn	Ni	Ba	Cr	Mn
unit	µg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	µg/kg	µg/kg
Med	0.12	27.1	11.5	32.7	0.45	1.64	0.27	25.2	1.32	0.22	0.22	0.29	0.19	48.4	40.8
Min	0.03	4.09	0.48	26.45	0.01	0.12	0.14	0.73	0.56	0.07	0.17	0.10	0.12	30.6	9.64
Max	0.58	47.4	19.5	35.6	0.13	28.4	4.91	63.5	1.82	0.47	0.29	0.55	0.34	59.7	226
SD	0.18	14.1	6.06	2.97	0.04	9.50	1.41	18.6	0.40	0.13	0.03	0.18	0.09	7.96	60.8
CV	92.9	56.5	52.6	9.37	98.9	153	214	72.2	31.8	55.3	14.7	71.7	65.8	17.4	102
Element	Ti	V	Ga	Ge	As	Se	Rb	Cd	Co	In	Sn	Sb	Te	Tl	Pb
unit	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/L	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg
Med	5.02	0.72	6.66	0.09	1.24	11.1	6.54	0.94	0.01	1.63	14.7	0.79	0.58	0.01	31.0
Min	0.50	0.32	0.50	0.05	0.30	5.60	2.13	0.57	0.01	0.36	7.17	0.49	0.15	0.09	3.31
Max	11.3	8.55	15.5	1.21	5.00	23.2	13.5	1.69	6.20	10.83	24.64	3.90	2.11	0.20	92.56
SD	3.21	2.47	3.99	0.33	1.45	4.48	3.48	0.30	1.86	3.57	5.85	1.25	0.54	0.06	24.7
CV	59.9	154	57.3	159	84.9	36.5	50.6	32.4	267	97.0	39.3	91.3	76.4	163	74.7

Studying the multi-elements content, in order to detect tendencies in the oil samples between varieties, principal components analysis was used. Promising groupings were observed using a model with two principal components and retaining 82.3% of the variance.

## Principal components analysis

